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(54) **PROCESS FOR PRODUCING CRYSTALLINE MALTITOL**

(57) There is provided methods for producing crystalline maltitol without using seed crystals, with good workability, at low costs, and with efficiency in a short time.

One of the methods comprises the steps of feeding a first maltitol aqueous solution to a kneader so as to knead and cool it, then feeding a second maltitol aqueous solution having a high solid content in place of the first maltitol aqueous solution and further kneading and

cooling the resulting mixture so as to produce a plastic mass, cooling the plastic mass so as to solidify it, and pulverizing the solidified mass. The other method comprises the steps of feeding a maltitol aqueous solution to a kneader so as to knead and cool it, then adding water and further kneading and cooling the mixture so as to produce a plastic mass, cooling the plastic mass so as to solidify it, and pulverizing the solidified mass.

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## Description

### Technical Field

**[0001]** The present invention relates to a production method of crystalline maltitol.

### Background Art

**[0002]** Maltitol is hardly digested and absorbed in digestive tracts and is hardly fermented by intraoral germs. Hence, it is used as a sweetener for low calorie foods, diet foods, low cariosity foods, diabetics and other applications. However, maltitol has a problem that since dried maltitol is significantly liable to absorb moisture and undergo deliquescence and therefore does not become powdery easily, it is inconvenient to handle.

**[0003]** To solve this problem, a number of techniques have been proposed for crystallization or pulverization of maltitol. Japanese Patent Publication No. 7-14953 proposes a method of producing a maltitol crystalline mixture solid by feeding an aqueous solution of maltitol continuously to an extruder having a long, narrow cooling and kneading zone, cooling and kneading the solution in the presence of seed crystals so as to produce maltitol magma, and extruding the maltitol magma continuously from an extrusion nozzle.

**[0004]** However, the production method using seed crystals has a problem that to improve a production rate, the amount of the seed crystals must be increased, and therefore a large amount of additional seed crystals must be prepared, thereby making a production process complicated.

### Disclosure of the Invention

**[0005]** An object of the present invention is to provide a novel production method of crystalline maltitol with significantly improved production efficiency as compared with that of the conventional production method of a maltitol crystalline mixture solid.

**[0006]** Another object of the present invention is to provide novel methods for producing crystalline maltitol without using seed crystals, with good workability, at low costs, and with efficiency in a short time.

**[0007]** Other objects and advantages of the present invention will be apparent from the following description.

**[0008]** According to the present invention, firstly, the above objects and advantages of the present invention are achieved by a method for producing (hereinafter occasionally referred to as "first invention") of crystalline maltitol which comprises the steps of feeding a first maltitol aqueous solution to a kneader so as to knead and cool it, feeding a second maltitol aqueous solution having a higher solid content than that of the first maltitol aqueous solution to the kneader so as to knead and cool continuously the resulting mixture and thereby produce a plastic mass, cooling the plastic mass so as to solidify

it, and pulverizing the solidified mass.

**[0009]** Further, according to the present invention, secondly, the above objects and advantages of the present invention are achieved by a method for producing (hereinafter occasionally referred to as "second invention") of crystalline maltitol which comprises the steps of feeding a maltitol aqueous solution to a kneader so as to knead and cool it, adding water thereto, further kneading and cooling continuously the mixture so as to produce a plastic mass, cooling the plastic mass so as to solidify it, and pulverizing the solidified mass.

### Preferred Embodiment of the Invention

**[0010]** Hereinafter, the methods of the present invention will be described in detail.

**[0011]** In the first invention, to produce crystalline maltitol, at least two types of raw materials having different solid contents are used. More specifically, as the first maltitol aqueous solution (hereinafter referred to as "first raw material") which is fed to a kneader first, a raw material having a relatively low solid content is used, and as the second maltitol aqueous solution (hereinafter referred to as "second raw material") which is fed to the kneader subsequently to the first raw material, a raw material having a higher solid content than that of the first raw material is used. Thereby, it becomes possible to omit a conventionally commonly practiced step of preparing additional seed crystals and feeding the seed crystals continuously to the kneader. Although the solid contents of the first and second raw materials are not limited to particular solid contents, it is important that the first raw material has a lower solid content than the second raw material. For example, as the first raw material, a maltitol aqueous solution with a solid content of 70 to 97 wt% and a purity of not lower than 88 wt% is preferred, and a maltitol aqueous solution with a solid content of 93 to 96 wt% and a purity of not lower than 90 wt% is more preferred. Meanwhile, as the second raw material, a maltitol aqueous solution with a solid content of 97.5 to 99.5 wt% and a purity of not lower than 88 wt% is preferred, and a maltitol aqueous solution with a solid content of 98 to 99 wt% and a purity of not lower than 90 wt% is more preferred.

**[0012]** The feed rates of the first and second raw materials vary according to the type and capacity of a kneader used and must therefore be set as appropriate. The feed rates of the first and second raw materials should be set such that optimum production efficiency is achieved in each kneader. Further, the feed rates of the first and second raw materials do not have to be the same, and they may be fed as different rates. For example, when a KRC kneader S2 type (product of KURIMOTO LTD.) is used as a kneader, the feed rate of the first raw material is preferably about 0.1 to 6.0 kg/hr, and the feed rate of the second raw material is preferably about 0.1 to 15 kg/hr.

**[0013]** Further, the jacket temperature of the kneader

at the time of feeding of the raw materials should be set at a temperature at which generated heat of crystallization can be removed. When a KRC kneader S2 type (product of KURIMOTO LTD.) is used, it is preferably not higher than 110°C, more preferably not higher than 95°C. This jacket temperature may be set at the time of feedings of the first and second raw materials according to the degree of generation of heat of crystallization.

**[0014]** The temperatures at which the raw materials are fed to the kneader are preferably temperatures at which maltitol crystals are not deposited. In consideration of the fact that higher flowability makes handling easier and ease of adjustment in forming a plastic mass, the temperature for the first raw material is preferably about 90 to 120°C, and the temperature for the second raw material is preferably 120 to 140°C.

**[0015]** In the first invention, switching from the first raw material to the second raw material is preferably performed after production of a white, opaque plastic mass is observed inside the kneader or at the outlet of the kneader after the first raw material is put in the kneader. This switching causes an improvement in the production efficiency of crystalline maltitol.

**[0016]** In the second invention, there is no need to use two types of raw materials as in the first invention, and crystalline maltitol can be produced with only one type of raw material. In that case, as the raw material, a maltitol aqueous solution with a solid content of 97.5 to 99.5 wt% and a purity of not lower than 88 wt% is preferred, and a maltitol aqueous solution with a solid content of 98 to 99 wt% and a purity of not lower than 90 wt% is more preferred.

**[0017]** The feed rate of the maltitol aqueous solution used as the raw material is the same as that of the first raw material in the first invention. To further improve the production efficiency of crystalline maltitol, it is possible to change the feed rate of the raw material to the same rate as that of the second raw material in the first invention after a step of adding water which will be described later. Switching between the feed rates of the raw material is preferably performed after production of a white, opaque plastic mass is observed inside the kneader or at the outlet of the kneader.

**[0018]** In the second invention, as described above, water is added after the raw material is fed. The water may be service water, for example. However, to produce pure, clean crystal maltitol, ion exchanged water is preferably used.

**[0019]** Water is preferably added in an amount of about 1.0 to 7.0 parts by weight based on 100 parts by weight of the maltitol aqueous solution which is a raw material. Water can be added several times to 10 times plus a few more times according to the extent of production of crystalline maltitol. Further, water can be charged into the kneader continuously or intermittently by use of a pump or the like. Timing of addition of water differs depending on a kneader used. Water should be added after discharge of the raw material solution from the out-

let is observed after the raw material is fully dispersed in the kneader.

**[0020]** Further, the jacket temperature of the kneader is the same as that in the first invention. However, it may be changed before or after the step of adding water.

**[0021]** In the present invention, the raw material is prepared by concentrating a maltitol aqueous solution having a solid content of about 70 wt%. In this case, the maltitol aqueous solution is preferably concentrated under a reduced pressure because the solution may be colored when it is heated under normal pressure so as to remove water. Since use of a colored raw material may interfere with production of target crystalline maltitol, it is important to prepare as colorless and transparent a raw material as possible.

**[0022]** A kneader usable in the present invention is not particularly limited and may be any of open, closed, batch and continuous kneaders as long as it is capable of performing kneading and cooling simultaneously. The kneader is preferably one capable of discharging a product continuously from the outlet after completion of kneading and cooling. Illustrative examples of such a kneader include an extruder, a continuous kneader, a mixtron and a kneadex. Of these, the extruder is more preferably used. Specific examples of the extruder include extruders such as a KRC kneader (product of KURIMOTO Ltd.), a twin screw extruder for food (product of JSW), and a twin screw cooking extruder (product of W & P Co., Ltd., Germany).

**[0023]** When a plastic mass is discharged from a continuous kneader, it can be in any form such as a noodle, a ribbon, a stick or a plate. In consideration of subsequent steps such as cooling and pulverization, the plastic mass is preferably discharged in the form of a noodle or ribbon. In that case, as a punching plate disposed at the outlet, one having a pore diameter of about 2 to 5 mm and a porosity of about 10 to 40% is preferably used.

**[0024]** A cooling method is not particularly limited. For example, a method comprising blowing cool air directly against a plastic mass discharged from a kneader, a method comprising leaving the mass to stand at room temperature or a method comprising cooling the mass to about room temperature on a metal mesh belt by cool air can be employed.

**[0025]** The obtained crystalline maltitol can be formed into powder or granules by pulverizing it without a drying step. A method of pulverization or granulation is not particularly limited. A commonly used pulverizer or granulator can be used. Further, if necessary, the obtained powder or granules may be dried by a commonly practiced drying method.

**[0026]** Hereinafter, the present invention will be described in more detail with reference to Examples and Comparative Examples.

## Examples

## Example 1

**[0027]** When a maltitol aqueous solution (purity = 90 wt%, solid content = about 95 wt%, 110°C, colorless and transparent) as a first raw material was continuously fed to a continuous kneader having a long, narrow kneading and cooling zone (KRC KNEADER S2 TYPE, product of KURIMOTO LTD., 60 rpm, jacket temperature = 80°C) at a rate of 4 kg/h and continuously kneaded and cooled, discharge of a white, opaque plastic mass from a punching plate at the outlet was observed after a while. Then, when another maltitol aqueous solution (purity = 90 wt%, solid content = about 98 wt%, 125°C, colorless and transparent) as a second raw material was continuously fed at a rate of 4 kg/h, a plastic mass in the form of a noodle was continuously discharged from the punching plate at the outlet. Then, although the feed rate of the second raw material was adjust at 8 kg/hr and the jacket temperature was adjust at 55°C, the plastic mass in the form of a noodle was discharged stably. This plastic mass was then cooled to 30°C or lower under blowing air so as to be solidified and then fed to POWERMILL P-3 TYPE (product of DALTON CO., LTD.) at 200 kg/hr so as to be pulverized. Thereby, crystalline maltitol of good quality was obtained.

## Example 2

**[0028]** A maltitol aqueous solution (purity = 92 wt%, solid content = about 97.6 wt%, 130°C, colorless and transparent) as a raw material was continuously fed to a continuous kneader having a long, narrow kneading and cooling zone (KRC KNEADER S2 TYPE, product of KURIMOTO LTD., 60 rpm, jacket temperature = 80°C) at a rate of 4 kg/h. After 5 minutes from the start of feeding of the raw material, ion exchanged water was added at a rate of 0.12 kg/hr, and the mixture was continuously kneaded and cooled. As a result of stopping addition of water when discharge of a white, opaque plastic mass from a punching plate at the outlet was observed, a plastic mass in the form of a noodle was continuously discharged from the punching plate at the outlet. Then, although the feed rate of the raw material was adjust at 8 kg/hr and the jacket temperature was adjust at 55°C, the plastic mass in the form of a noodle was discharged stably. This plastic mass was then cooled to 30°C or lower under blowing air so as to be solidified and then fed to POWERMILL P-3 TYPE (product of DALTON CO., LTD.) at 200 kg/hr so as to be pulverized. Thereby, crystalline maltitol of good quality was obtained.

**[0029]** As described above, according to the methods of the present invention, no seed crystals are used, and therefore crystalline maltitol can be produced with good workability, at low costs and with efficiency in a short time.

## Claims

1. A method for producing crystalline maltitol, comprising the steps of feeding a first maltitol aqueous solution to a kneader so as to knead and cool it, then feeding a second maltitol aqueous solution having a higher solid content than that of the first maltitol aqueous solution to the kneader and further kneading and cooling the resulting mixture so as to produce a plastic mass, cooling the produced plastic mass so as to solidify it, and pulverizing the solidified mass.
2. The method of claim 1, wherein the first maltitol aqueous solution has a solid content of 70 to 97 wt%, and the second maltitol aqueous solution has a solid content of 97.5 to 99.5 wt%.
3. A method for producing crystalline maltitol, comprising the steps of feeding a maltitol aqueous solution to a kneader so as to knead and cool it, then adding water to the kneader and further kneading and cooling the mixture so as to produce a plastic mass, cooling the produced plastic mass so as to solidify it, and pulverizing the solidified mass.
4. The method of claim 3, wherein the maltitol aqueous solution has a solid content of 97.5 to 99.5 wt%.
5. The method of claim 3, wherein water is added several times.
6. The method of claim 1 or 3, wherein the kneader is a continuous kneading-extruding machine having a cooler.

## INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP03/07613

| <b>A. CLASSIFICATION OF SUBJECT MATTER</b><br>Int.Cl <sup>7</sup> C07H15/04, 1/00<br><br>According to International Patent Classification (IPC) or to both national classification and IPC                                                                                                                                                                                                                                                                                                                                                                                                                |                                                                                                                                                                                       |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          |
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| <b>B. FIELDS SEARCHED</b><br>Minimum documentation searched (classification system followed by classification symbols)<br>Int.Cl <sup>7</sup> C07H1/00-1/08, 15/04-15/10<br><br>Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched<br><br>Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)<br>Caplus (STN), WPI (DIALOG)                                                                                                                           |                                                                                                                                                                                       |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          |
| <b>C. DOCUMENTS CONSIDERED TO BE RELEVANT</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |                                                                                                                                                                                       |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          |
| Category*                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 | Citation of document, with indication, where appropriate, of the relevant passages                                                                                                    | Relevant to claim No.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    |
| A                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         | JP 9-132587 A (Towa Chemical Industry Co., Ltd.),<br>20 May, 1997 (20.05.97),<br>Particularly, example 2, refer to the paragraph<br>of "Continuous Crystallization"<br>(Family: none) | 1-6                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |
| A                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         | JP 6-234786 A (Towa Chemical Industry Co., Ltd.),<br>23 August, 1994 (23.08.94),<br>Particularly, examples 1, 2<br>(Family: none)                                                     | 1-6                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |
| A                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         | EP 937733 A2 (KABUSHIKI KAISHA HAYASHIBARA<br>SEIBUTSU KAGAKU KENKYUJO),<br>25 August, 1999 (25.08.99),<br>& JP 11-217395 A & CN 1239099 A<br>& US 6120612 A & TW 429261 A            | 1-6                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |
| <input checked="" type="checkbox"/> Further documents are listed in the continuation of Box C. <input type="checkbox"/> See patent family annex.                                                                                                                                                                                                                                                                                                                                                                                                                                                          |                                                                                                                                                                                       |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          |
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| Date of the actual completion of the international search<br>16 September, 2003 (16.09.03)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                |                                                                                                                                                                                       | Date of mailing of the international search report<br>28 October, 2003 (28.10.03)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |
| Name and mailing address of the ISA/<br>Japanese Patent Office                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                            |                                                                                                                                                                                       | Authorized officer                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       |
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## INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP03/07613

## C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages                                                                      | Relevant to claim No. |
|-----------|---------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------|
| A         | EP 202165 A1 (ROQUETTE FRERES) ,<br>20 November, 1986 (20.11.86) ,<br>& EP 202165 B1 & FR 2581999 A<br>& DE 3663143 G & JP 61-268696 A<br>& JP 7-630 B2 | 1-6                   |

Form PCT/ISA/210 (continuation of second sheet) (July 1998)